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DIELECTRIC PROPERTIES OF EPOXY COMPOSITES WITH NANODISPERSED COPPER, CARBON MULTILAYER NANOTUBES AND COBALT WHEN MODIFYING THE ESHI BINDER

Petro STUKHLIAK, Doctor of Technical Sciences, Professor
Oleh TOTOSKO, Candidate of Technical Sciences, Associate Professor
Danylo STUKHLIAK, Candidate of Technical Sciences, Associate Professor
Ternopil Ivan Pulyu National Technical University

СТУХЛЯК Петро Данилович, д.т.н., професор
ТОТОСЬКО Олег Васильович, к.т.н., доцент
СТУХЛЯК Данило Петрович, к.т.н., доцент
Тернопільський національний технічний університет імені Івана Пулюя

The paper presents the results of a study of molecular mobility in an epoxy binder containing nanofillers of various types: nanodispersed copper (Cu), cobalt (Co), and carbon multilayer nanotubes (CMLN) under modification by electric spark hydro-impact (ESHI). The research focuses on the dielectric characteristics of the formed composites, in particular on the dielectric loss tangent, which serves as an informative indicator of segmental mobility of polymer chains and the intensity of relaxation processes in the solid state. It has been established that the introduction of nanofillers into the epoxy matrix leads to a systematic shift of the temperature maximum of the dielectric loss tangent and to a change in the shape of relaxation peaks, which indicates a modification of the molecular mobility of macromolecular fragments and the formation of interphase layers with altered energy states. Comparative analysis of the influence of Cu, Co, and CMLN reveals different efficiencies in the formation of interfacial interactions, determining the nature of dipole relaxation, the degree of restriction of segmental motion, and the polarization behavior of the composite. Carbon multilayer nanotubes demonstrate the most pronounced effect on relaxation processes due to the formation of a branched conductive network and barrier layers, whereas metallic nanoparticles mainly influence local polarization mechanisms associated with surface defects and electronic polarization. The obtained results confirm that ESHI modification combined with nanofilling enables controlled tuning of dielectric and relaxation properties of epoxy systems. This creates prerequisites for the development of functional polymer composites with predetermined electrical, polarization, and operational characteristics for applications in electrical engineering, electronics, and protective coatings.

Key words: *epoxy binder, nanofillers, dielectric losses, molecular mobility, carbon nanotubes, metal nanoparticles.*

Fig. 3. Table. 2. Ref. 16.

1. Problem formulation

The current development of polymer materials is aimed at creating multifunctional composites capable of combining high mechanical strength, thermal stability, resistance to aggressive environments, and improved electrophysical characteristics. One of the most promising areas of polymer modification is the use of various types of nanofillers, which cause significant changes in the structure, curing kinetics, and physicochemical properties of the polymer binder even at low concentrations [1]. Particular attention is paid to epoxy resins, which, due to their high adhesive ability, mechanical strength, chemical inertness and dielectric properties, are widely used in radio electronics, aerospace technology, electrical insulation coatings and structural composites.

It is known that the dielectric properties of polymers, in particular the tangent of the dielectric loss angle, are closely related to the molecular mobility of macrochains and the nature of relaxation processes in the solid state [2–6]. Studying changes in the loss tangent as a function of temperature or frequency allows us to assess the level of segmental mobility and the degree of restriction of dipole movements in the matrix. In the case of epoxy binders modified with nanodispersed additives and electric spark hydro-impact (ESHI), this indicator is sensitive to the presence of interphase interactions, the aggregate state of the filler and its effect on the microstructure of the polymer [2, 7].

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The problem lies in the lack of detailed research results on the patterns of influence on molecular mobility in the polymer matrix of fillers of various nature, both metallic (Cu, Co) and carbon (multilayer nanotubes). Despite a significant number of studies devoted to the influence of nanoparticles on the mechanical and thermal properties of epoxy composites, the issue of changes in dielectric parameters and, accordingly, the nature of intramolecular relaxation processes remains insufficiently covered. This is due to both differences in the morphology of nanofillers and the complexity of the formation of interphase layers, where the main processes of stress redistribution and changes in the potential energy of mobile segments occur.

2. Analysis of recent research and publications

The introduction of copper and cobalt nanoparticles into the epoxy binder can potentially affect the polarisation ability of the system due to the presence of surface defects involved in local orientation and electronic polarisation processes [8, 9]. At the same time, carbon multilayer nanotubes (CMLNs) form a branched conductive network in the polymer matrix, which can cause changes in the relaxation behaviour of the material due to the formation of barrier layers and new charge transfer paths. The combined action of metal and carbon nanofillers creates conditions for the emergence of effects that manifest themselves in a change in the temperature position of the dielectric loss tangent maxima, the broadening of relaxation peaks, and a change in dielectric permeability over a wide frequency range.

Currently, there is no comprehensive model describing the relationship between the nature of the nanofiller, its dispersibility, concentration, and the dielectric characteristics of the epoxy matrix. Most of the known results are fragmentary and obtained for systems with a limited set of parameters, which complicates the comparison and generalisation of data. In this context, a systematic study of the influence of nanodispersed Cu, CMLN and Co particles on the molecular mobility and polarisation properties of the epoxy binder based on the analysis of the temperature dependencies of the dielectric loss tangent is relevant.

For experimental studies, an ED-20 epoxy binder (dianic epoxy resin) was used, which is characterised by high adhesive properties, chemical resistance and low shrinkage during product formation. The epoxy binder was plasticised with diethylene glycol (DEG-1). Polyethylene polyamine (PEPA) was used as a hardener, which ensures the formation of a three-dimensional polymer mesh structure at room temperature with subsequent heat treatment.

To modify the epoxy binder, nanofillers of various types were used: nanodispersed copper (Cu) with an average particle size of 40–60 nm; nanodispersed cobalt (Co) with an average particle size of 50–70 µm; carbon multilayer nanotubes (CMLN) with a diameter of 10–30 nm and a length of 5–15 µm.

The dielectric properties of composite materials were studied to assess the effect of nanofillers on the polarisation properties and molecular mobility of the epoxy binder. The main parameter determined experimentally was the tangent of the dielectric loss angle (tg δ), which reflects the ability of the material to accumulate and dissipate electrical energy under the action of an alternating electric field.

The measurements were carried out using an E7-14 AC bridge, which ensures high accuracy in determining dielectric parameters over a wide frequency range. The experiments were conducted at three control frequencies – 0.1, 1.0, and 10 kHz, which allowed us to track the frequency dependence of polarisation processes in the material. Temperature control was carried out to ensure stable measurement conditions and to exclude the influence of temperature fluctuations on the results.

The samples for research were made in the form of cylinders with a diameter of D = 30 ± 0.5 mm and a height of h = 5 ± 0.05 mm. The thickness deviation across the surface did not exceed 8%, which guarantees the uniformity of the electric field between the electrodes during measurements. The working surfaces of the samples were levelled to ensure reliable electrical contact with the measuring electrodes.

The research was conducted in accordance with the standard methodology described in [10]. Based on the experimental data obtained, dielectric losses (ε'') or loss factor were calculated using the ratio between the imaginary and real components of complex dielectric permeability in accordance with the methodological recommendations [11]. The obtained values of ε' and ε'' allowed us to construct temperature dependencies tg δ, which were used to evaluate the nature of relaxation processes in the studied samples.

The resistance of coatings was determined in accordance with the requirements of regulatory document [12] using the two-point contact method with a direct current measuring bridge. The capacitance of the samples was calculated according to the methodology [13], based on the geometric dimensions and dielectric permeability indicators. For each type of sample, the measurements were repeated at least three times, after which the average values of the parameters and statistical deviation were calculated.

The total error in measuring dielectric permeability did not exceed ±2%, and the loss tangent did not exceed ±5%, which ensures the reliability of the results obtained. Experimental control, temperature stabilisation, and verification of the reproducibility of the results made it possible to obtain reliable information for further analysis of the influence of nanofillers on the electrophysical properties of the studied compositions.

3. The purpose of the article

The aim of this work is to establish the patterns of influence on molecular mobility in an epoxy polymer system of nanofillers of different nature based on the study of temperature and frequency dependencies of the dielectric loss tangent angle when modifying the matrix by electric spark hydroimpact. The results obtained will deepen our understanding of the physicochemical mechanisms of the formation of dielectric properties of nanomodified epoxy systems and contribute to the development of a new generation of materials with controllable polarisation, relaxation and electrical conductivity parameters. From a practical point of view, this opens up prospects for the creation of polymer composites with increased stability of operational properties for electrical and anti-corrosion applications.

4. Results and discussion

As a result of the experiments, a set of data was obtained reflecting the influence of nanodispersed fillers on the molecular mobility and dielectric properties of the epoxy binder. The analysis revealed characteristic patterns of change in the tangent of the dielectric loss angle depending on the nature, size, and concentration of nanoparticles. The results obtained indicate a correlation between the microstructure of the modified polymer and its relaxation characteristics. It has been established that metal and carbon nanofillers have different effects on the dynamics of polarisation processes in the material. Generalisation of the experimental data obtained made it possible to identify the main trends and mechanisms of change in the electrophysical properties of nanomodified epoxy compositions.

A study was conducted on CM filled with dispersed and nanoparticles of various nature when modifying the matrix by electric spark hydroimpact (ESHI) on the properties of epoxy composite materials (CM). Figs. 1, 2, and 3 show the results of studies of dielectric losses from the temperature of CM containing various concentrations of fillers (nanodispersed copper (Cu), cobalt Co, carbon multilayer nanotubes (CMLN) after modification of the ESHI of epoxy resin, which was subsequently plasticised with aliphatic resin DEG-1 (10 wt.% per 100 wt.% ED-20). For comparison, the graphs also show the results of the curves of the dependence of the loss factor (ε″) on the temperature of CM containing a filler based on a plasticised but unmodified ESHI matrix. It has been experimentally established that, in general, three sections can be distinguished on the curves of the dependence of dielectric losses (ε″) on temperature. It is shown (Fig. 1) that the introduction of nanodispersed copper (10 wt.% per 100 wt.% of binder) into an unmodified ESHI matrix leads to the appearance of three maxima of the relaxation process of groups, segments and chains. Pretreatment of ESHI epoxy oligomer in a plasticised matrix followed by the introduction of filler at different concentrations shows a significant shift of the relaxation process maxima to the higher temperature range [14].

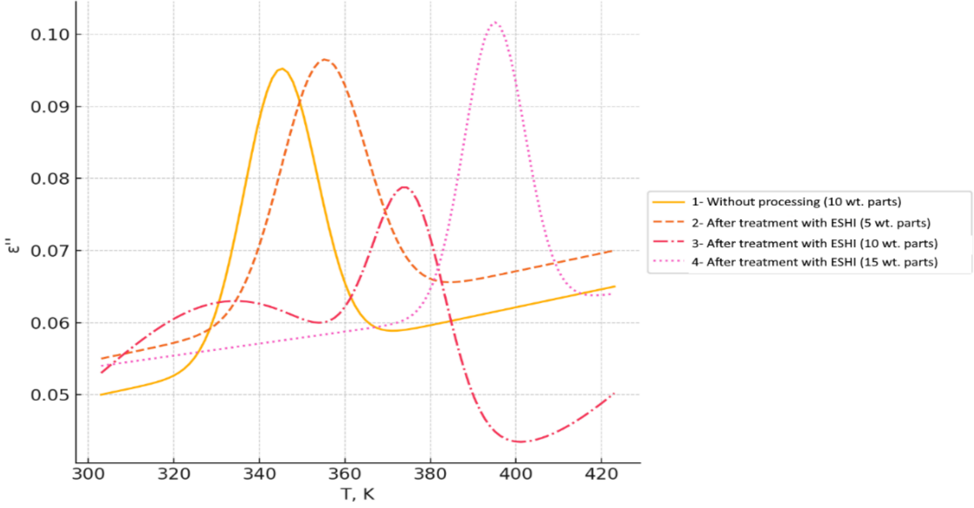

Fig. 1. Temperature dependence of dielectric losses (ε'') of epoxy composites containing nanodispersed Cu: 1 - without ESHI treatment (10 wt.%); 2 - after ESHI treatment (5 wt.%); 3 - after ESHI treatment (10 wt.%); 4 - after ESHI treatment (15 wt.%).

This indicates an improvement in the interaction at the interface between the ‘oligomer-dispersed filler’ phases. In our opinion, this is due to the activation of macromolecules and free radicals during the processing of ESHI. Such free radicals have greater activity and mobility compared to the initial macromolecules, which ensures significant interaction with active centres on the surface of copper particles. When filling the matrix with copper at a concentration of 5 mass parts per 100 mass parts of binder, this causes a significant increase in the maximum loss factor (ε″) to 0.02 (in the segment relaxation region) and to 0.03 (in the chain relaxation region). Such a sharp increase in the dielectric loss factor indicates high mobility in the surface layers of macromolecules. This indicates a low degree of cross-linking of the matrix around the filler and in the polymer volume, which is obviously associated with an insufficient concentration of dispersed particles in the system. Accordingly, to improve the cohesive characteristics of these CMs, the concentration of additives should be increased. It is shown (Fig. 1) that the introduction of 5...15 wt.% of nanodispersed copper into the matrix leads to a decrease in the maximum dielectric loss, i.e., to a decrease in mobility on the one hand, and to a more uniform layer-by-layer distribution of segments in the volume of the surface layers on the other. It should be noted that, in our opinion, the optimal concentration is a particle content of 10 mass parts per 100 mass parts of ED-20. A slightly different picture was observed when filling an untreated plasticised matrix with dispersed cobalt (Fig. 2).

It has been proven that the introduction of 40 mass parts of cobalt into the initial composition leads to the appearance of a single maximum of dielectric losses, which corresponds to the relaxation of segments in the adsorption layer. At the same time, the loss factor (ε″) of this maximum is quite high and amounts to 0.16. Modification of the ESHI epoxy oligomer with subsequent introduction of a filler at the same concentration leads to the appearance of three maxima of relaxation of segments and groups of macromolecules. However, it should be noted that the mobility of groups and segments in such layers is not high enough and amounts to ε″=0.05 (when analysing the relaxation process of the main chain). This indicates a sufficiently high degree of cross-linking in terms of the percentage of gel formation in the surface layers of CM with the specified filler. At the same time, the introduction of cobalt at concentrations of 30 and 50 mass parts per 100 mass parts of binder also causes a decrease in the loss factor and the manifestation of relaxation maxima (Fig. 2).

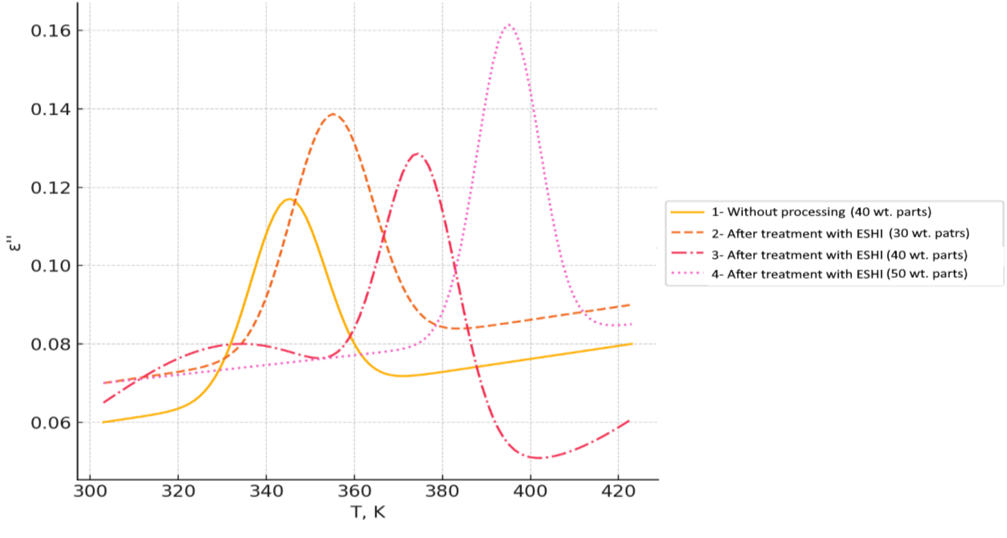

Fig. 2. Temperature dependence of dielectric losses (ε'') of epoxy composites containing nanodispersed Cu: 1 - without ESHI treatment (10 wt.%); 2 - after ESHI treatment (5 wt.%); 3 - after ESHI treatment (10 wt.%); 4 - after ESHI treatment (15 wt.%).

Analysis of the results of studies of the curves of dielectric loss dependence on temperature allows us to conclude that the optimal concentration of this filler in CM is 30–40 mass parts per 100 mass parts of binder. The introduction of cobalt at this concentration ensures a more uniform distribution of segments throughout the entire volume of the surface layer formed around the filler. An increase in the concentration of the filler in the system leads to a decrease in the width of the maxima, which, in turn, increases the mobility of macromolecules with increasing temperature.

From a scientific and practical point of view, it is interesting to study the relaxation processes of groups and segments in the surface layers of composites containing carbon multilayer nanotubes (CMLNs). It has been established (Fig. 3, Table 1) that the shift in the maximum temperature characteristic of the relaxation of groups in the second surface layer, depending on the filler concentration, was practically not observed and amounted to 395 K.

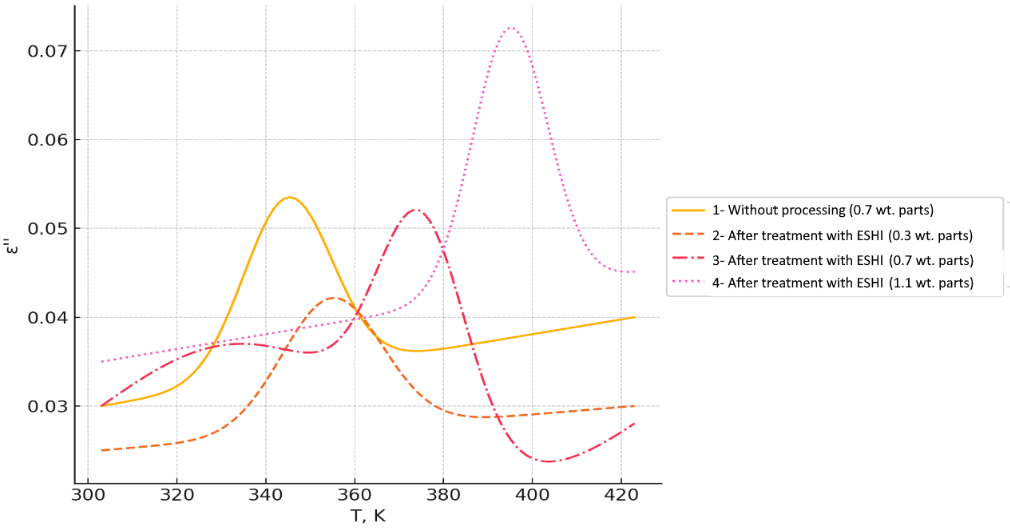

Fig. 3. Temperature dependence of dielectric losses (ε'') of epoxy composites containing carbon multilayer nanotubes: 1 - without ESHI treatment (0.7 wt.%); 2 - after ESHI treatment (0.3 wt.%); 3 - after ESHI treatment (0.7 wt.%); 4 - after ESHI treatment (1.1 wt.%).

At the same time, after modification of the ESHI matrix, a shift in the maximum temperature responsible for the relaxation of segments and macromolecules was observed in both directions along the temperature axis, depending on the concentration of dispersed particles. It is shown (Table 1) that at an additive concentration of 1.1 wt.%, the maximum temperature of the specified relaxation process shifts to the lower temperature region (by 20 K) after modification of the ESHI epoxy oligomer. The introduction of CMLN at a content of 0.3...0.7 mass % per 100 mass % of ED-20 causes a shift of the ε″ maximum to the higher temperature region (by 10 and 17 K, respectively). This is due to the overlap of the relaxation regions of segments and groups in the surface layers around the additives at the specified concentrations in the CM. Thus, such composites are characterised by a more uniform distribution of segments in the adsorption and surface layers around the filler in the composition. The introduction of fillers at higher concentrations leads to the formation of a material with a sufficiently rigid structure in the surface layers, especially in the adsorption layer [15].

The influence on the physicochemical interaction of the particle surface with the binder macromolecules has been proven. The optimal concentration of additives in the composite volume has been experimentally established. It should be noted that when evaluating these characteristics, both the width of the dielectric loss peak and its height on the ‘loss factor - temperature’ dependence curve should be taken into account. Only with such a comprehensive approach is it possible to assess the influence of surface activity and filler concentration on the degree of matrix cross-linking.

It is known [16] that physical modification, as well as chemical grafting of oligomers to the filler surface, improves physical interaction at the polymer-filler phase boundary. This, in turn, leads to a reduction in internal stresses in the surface layers around the filler. In this regard, it was interesting from a scientific and practical point of view to conduct research on molecular mobility in the surface layers around the filler. Such physical modification of dispersed particles will activate the physicochemical interaction of hydroxyl and other groups on the surface of additives blocked by hydrogen bonds. This, in turn, will improve interphase interaction, which, in our opinion, will manifest itself in the length and relative content of segments in the surface and adsorption layers.

It has been experimentally established that physical temperature modification of the dispersed filler affects molecular mobility in the surface layers in different ways (Table 2). In particular, it has been shown that temperature treatment of CMLN does not cause a shift in the maxima of dielectric losses during the relaxation process of groups. However, the maximum ε″ in the relaxation process of segments shifts to the region of higher temperatures by 20 K. It has been proven that such a sharp change occurs due to the activation of physical and mechanical processes after heat treatment of the filler, which is associated with the active influence of the surface of dispersed particles on the physicochemical interaction at the phase boundary ‘filler-polymer’. A different picture was observed after heat treatment of Cu and Co. In particular, when modifying Co, a decrease in the maximum dielectric loss, which is responsible for the relaxation process of groups, was observed. At this filler concentration (40 wt.% per 100 wt.% binder), after heat treatment of the filler, a tightly bonded material is formed in the surface layer, which is characterised by a significant extent. At the same time, the entire volume of the polymer transitions to the surface layer state.

Table. 1

Temperature characteristics of relaxation processes in initial and epoxy-modified composites

Filler	Mass. part.	Group relaxation process		Segment relaxation process		Circuit relaxation process	
		Process width, ΔT, К	Maximum temperature, T_m, К	Process width, ΔT, К	Maximum temperature, T_m, К	Process width, ΔT, К	Maximum temperature, T_m, К
Nanodispersed Cu (untreated)	10	10	345	15	375	18	433
Cu	5	12	355	14	375	17	395
Cu	10	14	375	18	395	20	410
Cu	15	16	395	20	410	21	420
Co (untreated)	40	16	345	18	375	20	395
Co	30	16	355	19	375	21	395
Co	40	20	375	21	395	23	405
Co	50	22	395	24	405	26	415
CMLN (untreated)	0.7	12	345	14	375	15	395
CMLN	0.3	10	355	12	375	14	395
CMLN	0.7	14	375	16	395	17	405
CMLN	1.1	16	395	18	410	20	420

In the case of heat treatment of copper, it has been established that the shift in the maximum temperatures after thermal modification of the filler occurs in the direction of lower temperatures on the time axis (when analysing the relaxation processes of segments). This, in turn, indicates the formation of a material with a high degree of cross-linking in the surface layer, but not as rigid as in the case of CM filling with cobalt. Accordingly, such composites are characterised by a less stressed state and better relaxation properties.

It has been proven that preliminary modification of fillers allows adjusting the properties of the material in the surface layers in accordance with the nature of the filler. In most cases, such modification ensures a more balanced state of the polymer in the surface layers, which contributes to the development of a method for predictable regulation of the operational properties of heterogeneous systems.

Table. 2

Temperature characteristics of relaxation processes in modified composites during preliminary heat treatment (T=423 K) of the filler

Filler	Heat treatment of fillers at 423 K	Group relaxation process		Segment relaxation process		Circuit relaxation process	
		Process width, ΔT, К	Maximum temperature, Tm, К	Process width, ΔT, К	Maximum temperature, Tm, К	Process width, ΔT, К	Maximum temperature, Tm, К
Cu	-	10	345	15	375	18	395
Cu	+	12	343	14	366	17	387
Cu	+	14	361	18	381	20	402
Cu	+	16	383	20	401	21	410
Co	-	16	345	18	375	20	385
Co	+	18	342	19	363	21	388
Co	+	20	363	21	384	23	397
Co	+	22	386	24	397	26	402
CMLN	-	12	332	14	375	15	395
CMLN	+	10	346	12	367	14	388
CMLN	+	14	364	16	388	17	400
CMLN	+	16	383	18	400	20	406

Note: + – heat treatment of filler; - – untreated filler.

At the same time, research results show the importance of conducting electric spark hydro-impact of epoxy oligomer followed by hydrodynamic alignment of components with a plasticiser (aliphatic resin DEG-1 and filler). This step-by-step creation of a matrix followed by the introduction of dispersed particles and a hardener ensures the formation of composites with high cohesive strength due to the creation of surface layers of considerable length and with a high degree of cross-linking.

5. Conclusion

Studies of dielectric losses have established that in composite materials at different stages of temperature increase, three areas of relaxation of groups, segments and chains of matrix macromolecules were observed, respectively. The obtained relaxation regions were interpreted through the analysis of the relaxation processes of different kinetic units of the epoxy binder, corresponding to their location on the temperature-time dependence curves of the dielectric loss tangent. This, in turn, allows for the predictable regulation of structure formation processes in the material by selecting the optimal filler concentration and temperature-time conditions for processing the epoxy composite. In our opinion, a promising approach is to regulate the structure in the surface layers around the filler by pre-heat treating it in a temperature field under specific conditions. Depending on the nature of the filler, this physical modification allows us to influence the length of the surface layers. In the future, we plan to conduct research to create materials with relatively homogeneous surface layers of considerable length.

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ДІЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ЕПОКСИДНИХ КОМПОЗИТІВ З НАНОДИСПЕРСНОЮ МІДДЮ, ВУГЛЕЦЕВИМИ БАГАТОШАРОВИМИ НАНОТРУБКАМИ ТА КОБАЛЬТУ ПРИ МОДИФІКАЦІЇ ЗВ'ЯЗУВАЧА ЕІГУ

У роботі наведено результати дослідження молекулярної рухливості епоксидного в'яжучого, що містить нанонаповнювачі різної природи: нанодисперсний мідь (Cu), кобальт (Co) та вуглецеві багатошарові нанотрубки (CMLN), за умов модифікування електроіскровим гідроударом (ESHI). Основну увагу зосереджено на аналізі діелектричних характеристик одержаних композитів, зокрема тангенса кута діелектричних втрат, який є інформативним показником сегментальної рухливості полімерних ланцюгів і інтенсивності релаксаційних процесів у твердому стані. Встановлено, що введення нанонаповнювачів в епоксидну матрицю зумовлює систематичний зсув температурного максимуму тангенса діелектричних втрат і зміну форми релаксаційних піків, що свідчить про модифікацію молекулярної рухливості фрагментів макромолекул та формування міжфазних шарів зі зміненими енергетичними станами. Порівняльний аналіз впливу Cu, Co та CMLN виявив різну ефективність формування міжфазних взаємодій, які визначають характер дипольної релаксації, ступінь обмеження сегментальних переміщень і поляризаційну поведінку композиту. Вуглецеві багатошарові нанотрубки демонструють найбільш виражений вплив на релаксаційні процеси завдяки формуванню розгалуженої провідної мережі та бар'єрних шарів, тоді як металеві наночастинки переважно впливають на локальні механізми поляризації, пов'язані з поверхневими дефектами та електронною поляризацією. Отримані результати підтверджують, що модифікування ESHI у поєднанні з нанонаповненням забезпечує кероване регулювання діелектричних і релаксаційних властивостей епоксидних систем. Це створює передумови для розроблення функціональних полімерних композитів із наперед заданими електричними, поляризаційними та експлуатаційними характеристиками для застосування в електротехніці, електроніці та захисних покриттях.

Ключові слова: *епоксидне в'яжуче, нанонаповнювачі, діелектричні втрати, молекулярна рухливість, вуглецеві нанотрубки, металеві наночастинки.*

Рис. 3. Табл. 2. Літ. 16.

INFORMATION ABOUT THE AUTHORS

Petro STUKHLIAK – Doctor of Technical Sciences, Professor of the Department of Computer-Integrated Technologies of Ternopil Ivan Pulyu National Technical University (St. Ruska, 56, Ternopil, Ukraine, 46000, e-mail: stukhlyakpetro@gmail.com, https://orcid.org/0000-0001-9067-5543).

Oleh TOTOSKO – Candidate of Technical Sciences, Associate Professor, Associate Professor of the Department of Computer-Integrated Technologies of Ternopil Ivan Pulyu National Technical University (St. Ruska, 56, Ternopil, Ukraine, 46000 e-mail: totosko@gmail.com, https://orcid.org/0000-0001-6002-1477).

Danylo STUKHLIAK – Candidate of Technical Sciences, Associate Professor, Associate Professor of the Department of Computer-Integrated Technologies of Ternopil Ivan Pulyu National Technical University (St. Ruska, 56, Ternopil, Ukraine, 46000 e-mail: e-mail: itaniumua@gmail.com, https://orcid.org/0000-0002-9404-4359).

СТУХЛЯК Петро Данилович – доктор технічних наук, професор кафедри комп'ютерно-інтегрованих технологій Тернопільського національного технічного університету імені Івана Пулюя (м. Тернопіль, вул. Руська, 56, e-mail: stukhlyakpetro@gmail.com, https://orcid.org/0000-0001-9067-5543).

ТОТОСЬКО Олег Васильович – кандидат технічних наук, доцент кафедри комп'ютерно-інтегрованих технологій Тернопільського національного технічного університету імені Івана Пулюя (м. Тернопіль, вул. Руська, 56, e-mail: totosko@gmail.com, https://orcid.org/0000-0001-6002-1477).

СТУХЛЯК Данило Петрович – кандидат технічних наук, доцент кафедри комп'ютерно-інтегрованих технологій Тернопільського національного технічного університету імені Івана Пулюя (м. Тернопіль, вул. Руська, 56,, e-mail: itaniumua@gmail.com, https://orcid.org/0000-0002-9404-4359).